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13. ABSTRACT (Maximum 200 words)

Liquid phase ion mobility spectrometry (LPIMS) is a novel analytical technique where ions are separated by electric field in a liquid medium. The electric field is established via a series of electrodes spaced evenly through the ion drift tube, similar to the drift tube design used in gas-phase IMS. Because no electrolyte is used in LPIMS, the method is a low current (i.e. low noise) method in which the current is carried by the ions produced in the ionization source.

This report documented the first LPIMS spectra of aqueous samples drifting through an organic liquid phase. LPIMS spectra of aqueous ammonium nitrate and aqueous sodium chloride solutions were obtained using two different LPIMS designs. A single peak was observed for sodium ion with a reduced mobility, K_0, of 5.3×10^{-4} cm^2 V^{-1} s^{-1}, which is similar to the expected value of 5.7×10^{-4} cm^2 V^{-1} s^{-1}.

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Figure 1 shows a schematic diagram of a liquid phase IMS instrument made from Teflon. The tube was made by producing half-cylinders on each half of a Teflon block. Slits were then cut into the block one mm apart and thin ion mobility drift rings were inserted in each slit. When the two halves were combined they form an ion mobility tube five mm wide and 20 mm long.

Non-aqueous solvents were pumped into the Teflon IMS to form the "drift liquid" while a needle was inserted in the opposite in of the tube to introduce the sample. At the end of the tube where the solvent was introduced was a faraday plate for ion detection.

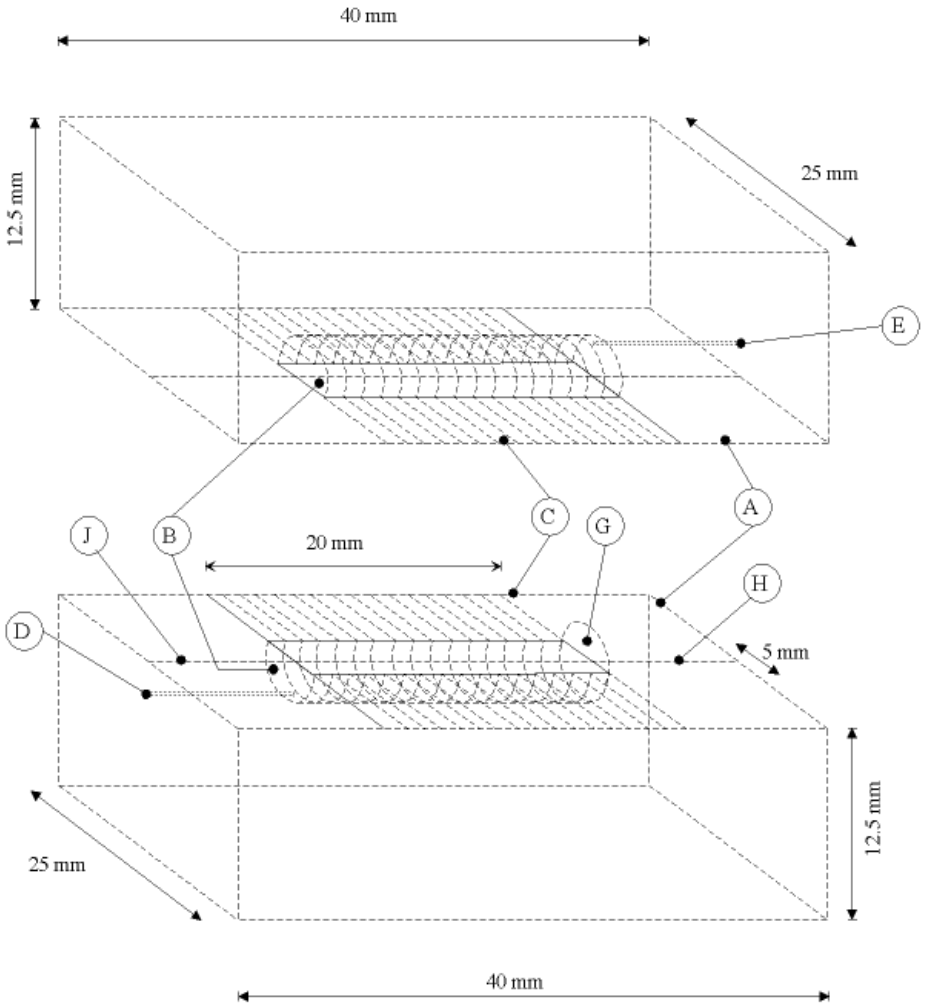

Figure 1: Liquid Phase IMS instrument made from

Figure 2 shows the voltage that was placed on each ring. On ring #1, the ring where the sample was introduced was held at 1000V and the voltage was dropped over 15 rings with ring #16 being the collector electrode. Thus, the field in this prototype was about 625 V/cm.

No ion gate was used with this prototype rather ring 5 was shorted to ring 7 to form a potential well through which the ions could not migrate. To open the gate, ring #5 was switched to its normal position in the resister chain and the trapped ions were injected into the drift region of the spectrometer.

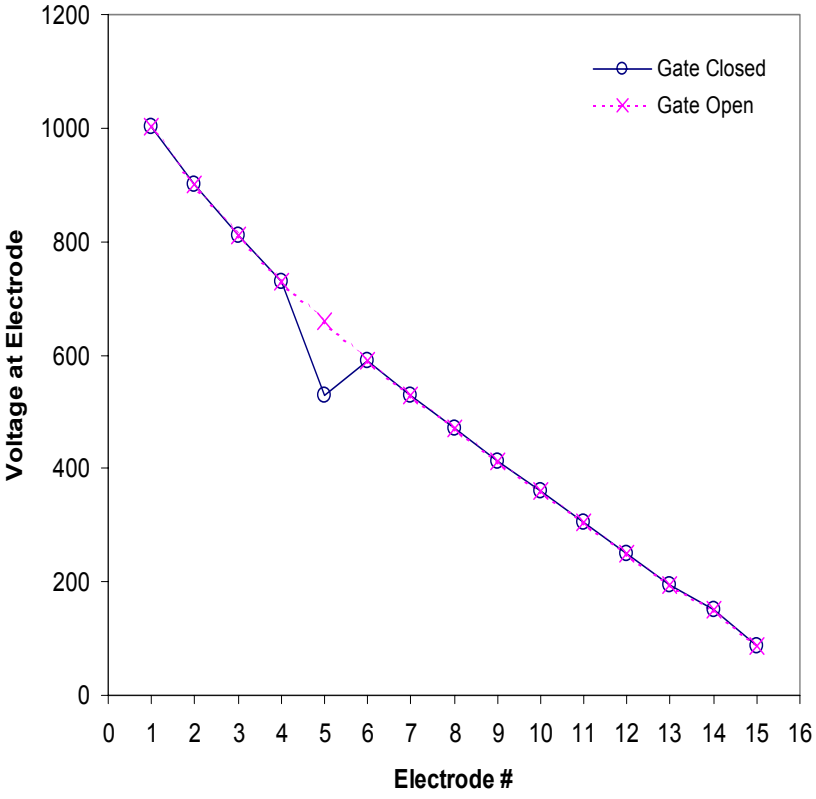

Figure 2: Voltage on each ring in the IMS.

Figure 3 shows the current (~650 nAmps) obtained at the collected electrode when an aqueous solution of 0.1M HCl was injected at 1 µL/min for 30 seconds into the liquid phase decanol. For these studies ring #5 was held at a potential, which would allow the ions to continuously pass through the tube.

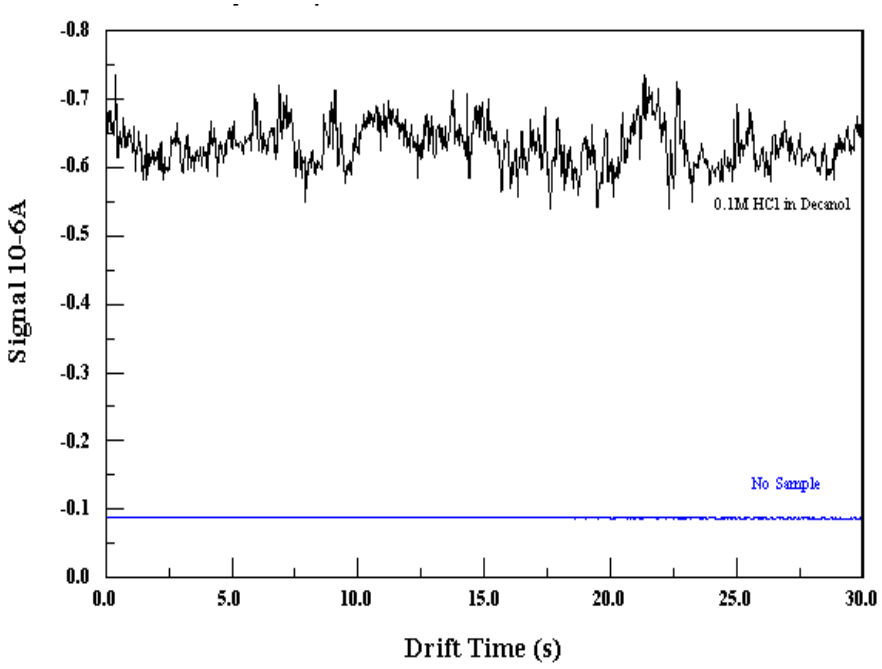

Figure 3: Ion signal for HCl in decanol with gate open.

Figure 4 and 5 show the first liquid phase ion mobility spectra obtained. While noisy and relatively low resolution, they do demonstrate that it is possible to achieve IMS in the liquid phase. Figure 4 is the IMS spectrum obtained when 0.25 μL/min of an aqueous solution of 50 ppm NH_4NO_3 was introduced into a liquid phase of mineral oil with no flow. The length of the drift tube for this run was 12.25 mm, which was the same as all of the runs using the Teflon tube. The gate was pulsed open for 5 seconds and 3 kV was placed on the first ring.

Figure 5, however, gives a signal peak for a 50ppm aqueous sample of NaCl, which was introduced into the mineral oil liquid phase at a rate of 1μL/min. The voltage on the needle was 1 kV and the gate pulse width was only 0.2 seconds.

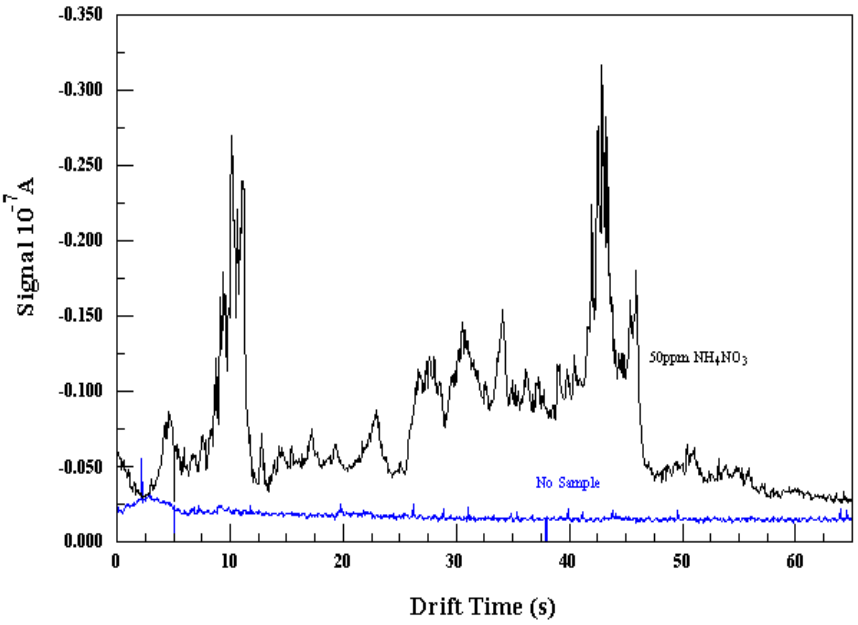

Figure 4: Liquid Phase ion mobility spectrum of NH_4^+.

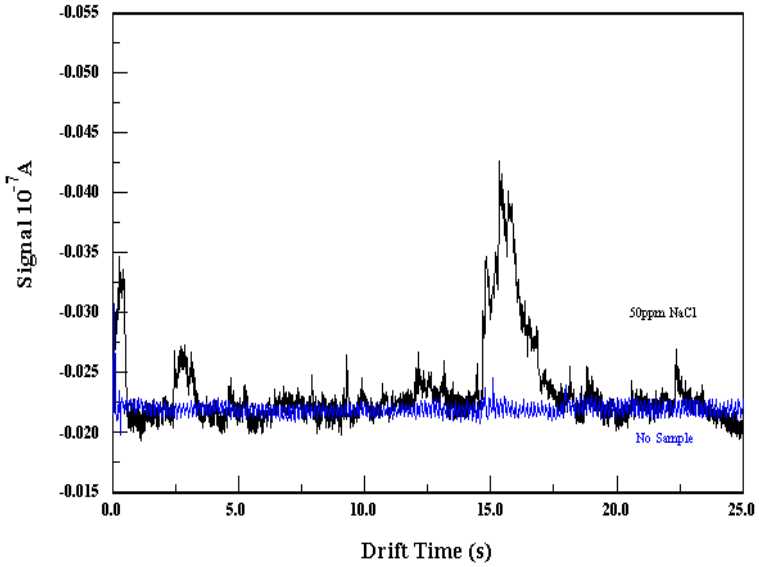

Figure 5: Liquid Phase IMS of Na^+

While these data were promising, data taken with a completely different IMS tube design looked even better. This design is shown in **Figure 6** and was constructed similar to gas phase IMS drift tubes although the dimensions approximately 1/10 of those used for gas phase ion mobility spectrometers.

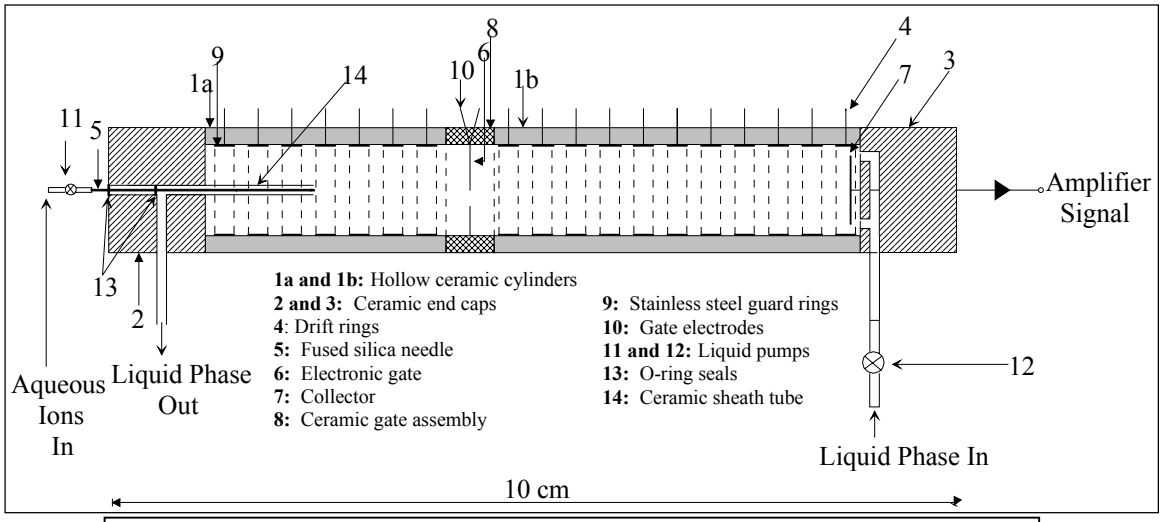

Figure 6: Liquid Phase IMS tube 1/10 size of Gas Phase Tube

A series of stainless steel rings (#9) connected with 1 MΩ resistors (#4) were used to create a homogeneous electric field throughout the tube. The liquid phase, mineral oil, was introduced (#12) at the collector electrode (#7) end of the tube though an insulated end cap (#3). The liquid phase flowed counter to the ion flow in the tube and exited the tube through a glass exit (#14) located in the ionization region of the spectrometer. The sample solution was introduced into the spectrometer via a liquid chromatographic type injection loop (#11). The sample solution was then pass into the ionization region of the spectrometer through an introduction needle (#5). Ions were extracted from the sample drops and directed through the liquid phase toward the ion gate (#8) by the electric field. Similar to the ion gates we construct for gas phase IMS, this liquid phase IMS consisted of a set of parallel wires (#6) in which each alternate wire (#10) was electrically isolated. Thus, we generate an electric field orthogonal to the drift tube field and stop the ions from migrating through the tube. Electronically we can pulse the gate open and allow a packet of ions to enter the ion separation region of the tube and continue to migrate toward the collector electrode. Both the ionization region of the tube and the separation region of the tube will be contained within alumina tubes (#1a and #1b).

Figure 7 shows two spectra, one with only water introduced and the other with 1ppm aqueous solution of NaCl. Injection volumes were 2 μL. For these experiments the drift length was 3.80 cm, the voltage on the first ring was 2 kV. The ion gate was a typical B-N gate, which was held open for 100 ms. The liquid phase was mineral oil introduced at a countercurrent flow of 1ml/min.

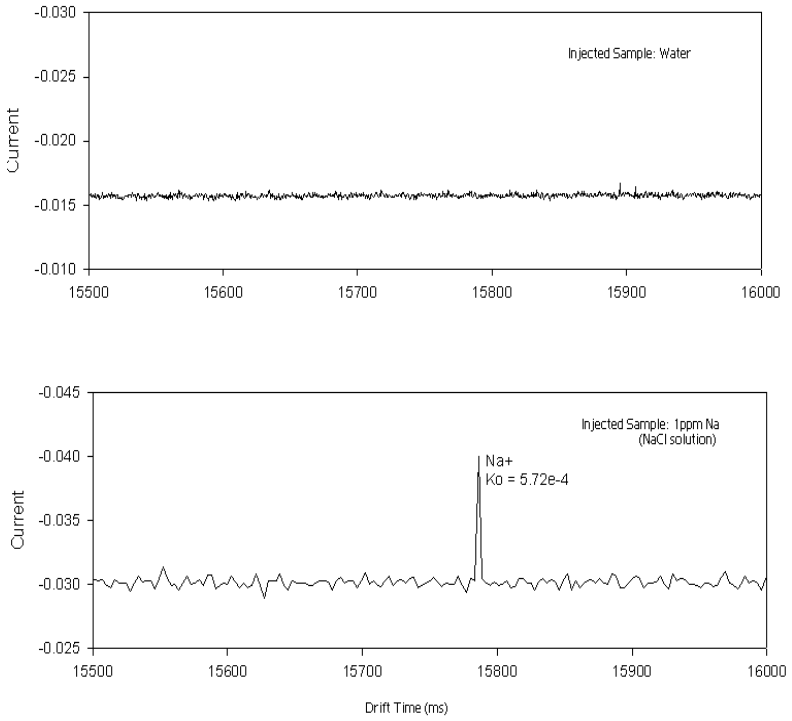

Figure 7: Liquid Phase IMS of pure water and 1ppm of NaCl

As can be seen from the spectrum, the Na^+ provided a single peak with a K_0 value (5.3×10^{-4} cm^2 V^{-1} s^{-1}) similar to that expected (5.7×10^{-4} cm^2 V^{-1} s^{-1}) for Na^+ in water.

While the Figures 4, 5, and 7 are encouraging, both of these prototype IMS instruments leaked liquid phase constantly, making the experiments messy, difficult to reproduce, and dangerous.